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A diesel-driven, metal-based solid oxide fuel cell

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HIGHLIGHTS

- Operation of a metal-based solid oxide fuel cell coupled with an diesel reformer.
- Analysis for stable operation in terms of thermodynamics and experiments.
- Modified condition is suggested in terms of oxygen partial pressure.
- A metal-based single stack with an area of 50-mm × 50-mm is successfully operated.
- Stable operation for 1000 h with a degradation rate of 4%/1000 h.

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ABSTRACT

This study examines the performance of a metal-based solid oxide fuel cell (SOFC) coupled with an integrated diesel fuel processor, with a focus on operating stability. The reformate is produced by an autothermal reformer (ATR), desulfurizer, and post-reformer using commercial diesel. Diesel reformate possesses the characteristics of low fuel concentration and high steam quantity due to its fuel processing condition for stable operation. These characteristics lead to high oxygen partial pressure at the anode which causes the oxidation of the cell's metallic component. Various approaches, such as thermodynamic calculation, temperature-programmed reduction (TPR) analysis, and the electrochemical performance of a single cell, are used to prove the oxidation phenomenon in this experiment. As a result, the key factors for stable operation are discovered, and a metal-based SOFC single stack with an area of 50-mm × 50-mm is successfully operated for 1000 h at a 4%/1000 h degradation rate under a modified condition of diesel reformate. To ensure the SOFC's stable operation using the reformate, it is recommended that the operating condition should be considered from a viewpoint combining fuel composition, oxygen partial pressure, and temperature. The modified condition for the long-term operation of a metal-based SOFC using diesel reformate is also suggested in this paper.

1. Introduction

Solid oxide fuel cells (SOFC) are a type of ceramic fuel cell that have become one of the most promising candidates as an alternative energy technology [1,2]. The advantages of SOFC include high efficiency, due to their high operating temperature, and fuel flexibility, as both hydrogen and carbon monoxide can be used as fuel, and even light hydrocarbons such as methane can be directly fed to the anode [3,4]. This flexibility makes the development of SOFC systems advantageous because the fuel supply system can be simpler. Given these advantages, there have been many efforts to complete and commercialize various SOFC systems, such as distributed power system and auxiliary power units (APU). A fuel process technology focused on liquid fuels such as gasoline and diesel has been developed to capitalize on the system's fuel flexibility. Diesel in particular has the most potential of any hydrocarbon to convert SOFC fuel because of its high energy density and well-built infrastructure, so it will be a prime candidate for commercialization once diesel reforming technology has been successfully developed. Although SOFC stack technology is the most important to developing SOFC systems, there are known difficulties associated with it, such as low mechanical strength and low sealing efficiency, also known as a dissimilar joining problem between a conventional cell and the metal interconnect. A metal-based SOFC has been suggested and developed to overcome these drawbacks and is fabricated with a thin coating of an electrode and electrolyte on a

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metal support [5]. There are still several problems associated with metal-based SOFC, such as the fabrication process and cathode sintering method, although the latter can compensate for the defects inherent in conventional SOFC.

The development of metal-based SOFC has been focused on the fabrication method, especially coating technologies such as colloidal spray techniques, aerosol spray deposition, suspension plasma spraying (SPS), pulsed laser deposition (PLD), and atmospheric plasma spraying (APS) [6–16]. Metal-support materials have also been spotlighted because SOFCs are operated at high temperatures, and metal-based SOFC can be more vulnerable to degradation by oxidation, so metal supports must be resistant to oxidizing atmospheres. In the same vein, there have been many efforts to reduce the operating temperature of metal-based SOFC. Instead of yttria-stabilized zirconia (YSZ), which is widely used as an electrolyte material, specialized materials for intermediate operating temperature such as Gd-doped ceria (GDC) and Mg-doped lanthanum gallate (LSGM) have been studied [17]. Cathode material selection and fabrication methods have also proven a challenge in metal-based SOFC because the cathode is normally sintered in an oxidizing atmosphere and high temperature of over 1000 °C. Metal-based cells encounter serious oxidation problems at the joint between the metal support and electrodes during the cathode sintering process. As a result, cathode materials have been studied for their potential to withstand in-situ sintering and sintering in a reducing atmosphere [18–20].

Diesel, a commonly used fuel in industry and the automobile market, contains a large amount of hydrogen that can be used for fuel cells. Diesel is basically a liquid fuel and thus must be evaporated when it is supplied to a reforming reactor. The evaporated fuel is reacted with steam, air, or both at over 800 °C on catalysts and finally converted to a reformate that can be used for SOFC. There are several methods used to reform diesel, including steam reforming (SR), partial oxidation (POx), and autothermal reforming (ATR). Steam reforming is an endothermic reaction that reacts with steam, so it is necessary to have a heating and water supply system that increases the total system size, although the process is still the most efficient. Partial oxidation is an exothermic reaction that reacts with air and has the advantages of rapid start-up, rapid reaction speed, and compact system size. However, there are significant problems associated with the process's long-term stability, such as catalyst degradation and acceleration of carbon deposition. Autothermal reforming (ATR) is slightly exothermic, which can be self-sustaining, and reacts with steam and air [21,22]. ATR is a combination reaction of SR and POx and possesses advantages of each method, so it is the most appropriate way to reform diesel stably [23–25]. Many groups have focused their efforts on the development of diesel reforming using ATR, and research and development is focused on catalysts, the oxygen to carbon ratio (OCR), the steam to carbon ratio (SCR), operating temperature and the desulfurizing method. Recently, an integrated diesel fuel processor has been developed to produce a fuel appropriate for SOFC that is composed of three different part: (1) ATR, (2) desulfurization, and (3) post-reforming [26]. Post-reforming is a process that removes low hydrocarbons such as ethylene, which can negatively affect SOFC cells [27,28]. The long-term operation of this processor has been validated for 2500 h, indicating that diesel reforming technology is ready for use in fuel cell systems [26].

SOFC and fuel processors have been studied mostly individually, but they should be studied together and systemized. The stability of SOFC operation when the fuel is supplied by a fuel processor has not been guaranteed, indicating that a study focused on the coupled simultaneous operation of an SOFC and reformer is necessary. However, a few problems have been anticipated in terms of gas composition when the developed diesel fuel processor is used. Diesel is converted to usable fuel using the ATR process, which requires both air and water. The final gas product has a relatively higher amount of steam and nitrogen, and generates a lower fuel concentration. This lower concentration leads to higher oxygen partial pressure and a lower open circuit voltage. In particular, the high oxygen partial pressure at the anode can cause oxidation of the anode and metallic component in the cell, which may result in performance degradation.

In this article, a metal-based SOFC that increased the cell's mechanical strength and sealing efficiency is operated using the reformate produced by the developed diesel fuel processor, which have been validated for 2500 h. The problems that arise during operation are analyzed and solved using thermodynamics and experiments. Co-operation between the SOFC and diesel reformer is necessary to develop such an SOFC system but has rarely been discussed and studied, especially from an operating condition viewpoint. This study concentrates on drawbacks arising from this co-operation and discusses the system's operating conditions.

2. Experimental

The diesel-driven SOFC system was installed according to the schematic diagram shown in Fig. 1. The integrated diesel fuel processor was directly connected to the SOFC stack and various gases were supplied to the stack. The gas lines were heated and insulated when the vaporized water flowed. Water was supplied using a HPLC pump and vaporized before being supplied to the SOFC anode. The reformate gas was produced by the integrated diesel fuel processor, which consisted of an autothermal reformer, a desulfurizer, and a post-reformer. Commercial diesel (GS-Caltex, South Korea) was used as fuel in the integrated fuel processor and was atomized using an ultrasonic injector to be supplied into the ATR reactor. The catalysts were Pt on Gd-doped CeO_2 (GDC) as an autothermal reformer, ZnO as a desulfurizer, and Ru on GDC as a post-reformer, and the operating temperatures of the three reactors were 800 °C, 400 °C, and 550 °C, respectively. To operate the ATR reactor, the steam to carbon ratio (SCR), oxygen to carbon ratio (OCR), and GHSV were 3, 0.8, and 12,500 h^{-1}, respectively [26]. In this study, synthetic gas, simulated as the reformate produced by the integrated diesel reformer, was also used for convenience in the experiments and analysis. Water was provided to the anode with the synthetic gas after vaporization.

The metal-based solid oxide fuel cell was fabricated by the high temperature sinter-joining process [29,30]. Each metal-based single cell was composed of a catalytic layer that included an anode and electrolyte, functional interface layer, and metal support. The catalytic layer and metal support bonded well with the functional interface layer, which was made up of metal powder and anode materials. The metal powder used in the functional interface layer was AISI 410, which consists mainly of iron and chromium. The metal support also used typical AISI 430 stainless steel. Two singe cell sizes were used: a round cell with a 25-mm diameter, called a button cell, and a square cell with area of 50-mm × 50-mm. The squared cell was fabricated as a single stack and directly connected with an interconnect created by laser welding to improve sealing efficiency. 8YSZ/NiO was used as an anode and 8YSZ as an electrolyte. $Gd_{0.1}Ce_{0.9}O_{1.9}$ (GDC, surface area of 46 $m^2\ g^{-1}$, Praxair, Inc.) was used as a buffer layer, while $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-d}$ (LSCF, surface area of 4.2 $m^2\ g^{-1}$, Fuel Cell Materials) and $Sm_{0.2}Ce_{0.8}O_{1.9}$ (SDC, surface area of 212 $m^2\ g^{-1}$, Fuel Cell Materials) were used as a composite cathode and mixed in proportions of 60 wt. % and 40 wt. %, respectively. The buffer layer and composite cathode were screen-printed and in-situ sintered at the operating temperature. The thicknesses of anode, electrolyte, buffer layer, and cathode were 1.5 mm, 20 um, 20 um, and 20 um, respectively.

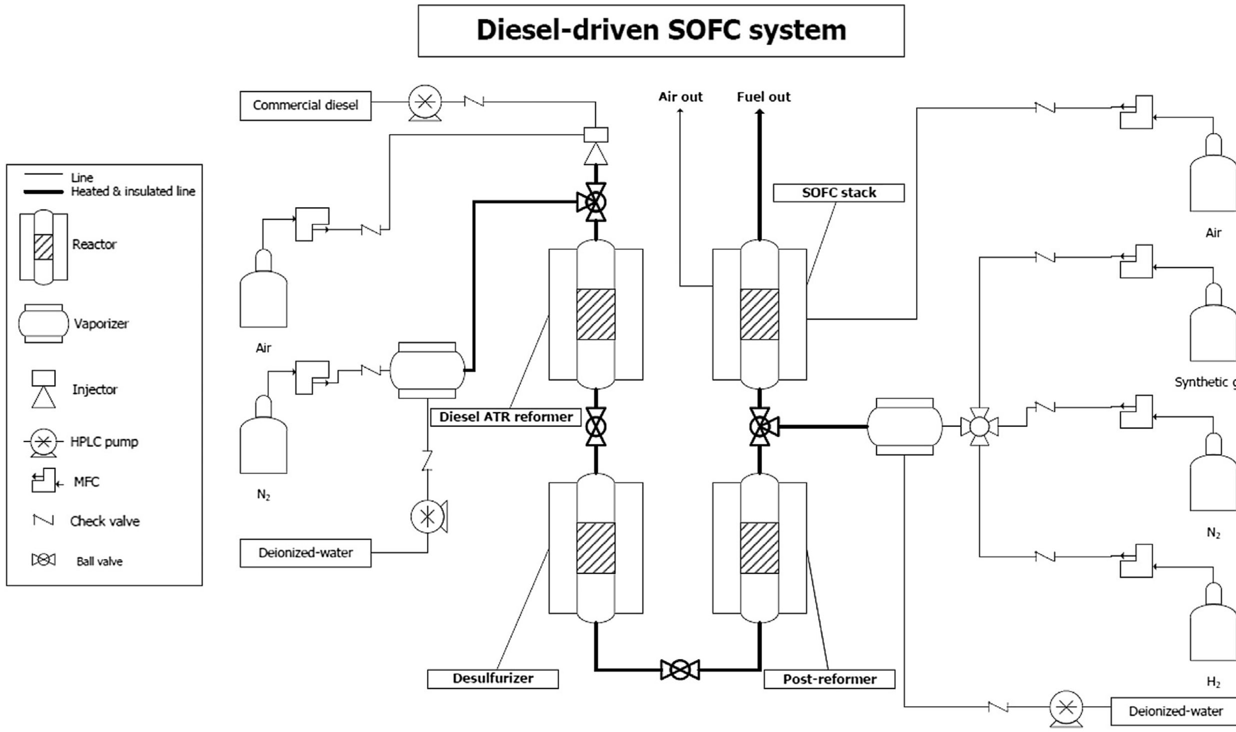

Fig. 1. Schematic diagram of the diesel-driven SOFC system.

Table 1
Gas compositions of diesel reformate and synthetic gas.

	Diesel reformate (%)	Synthetic gas (%)
Hydrogen (H_2)	14.95	22
Carbon monoxide (CO)	3.32	4.9
Carbon dioxide (CO_2)	9.53	14
Nitrogen (N_2)	39.69	58.4
Methane (CH_4)	0.47	0.7
Steam (H_2O)	32.04	–

An AC four-probe, two-electrode method was used to measure the current density–cell voltage–power density (*I*–*V*–*P*) and electrochemical ac impedances using a Solartron 1287/Solartron 1260 (electrochemical interface/impedance, gain-phase analyzer). A DC electronic load (Daegil, EL-1000P) was used to apply the current and analyze a single stack's long-term operation. The thermodynamic equilibrium partial pressures were calculated using HSC Chemistry for Windows, version 5.1.

3. Results

To ensure the SOFC stack's stable operation, low hydrocarbons above C2, which cause carbon deposition at the SOFC's anode, are removed by the post reformer, the composition of which is shown in Table 1. The integrated diesel fuel processor is successfully validated for 2500 h at a 5% degradation rate and is stable enough to use as a SOFC fuel for stable operation, although a small amount of light hydrocarbons are detected after 2000 h of operation because of catalyst degradation [26].

Hydrogen and diesel reformate are successively supplied as fuels. The hydrogen is humidified by bubbling at room temperature

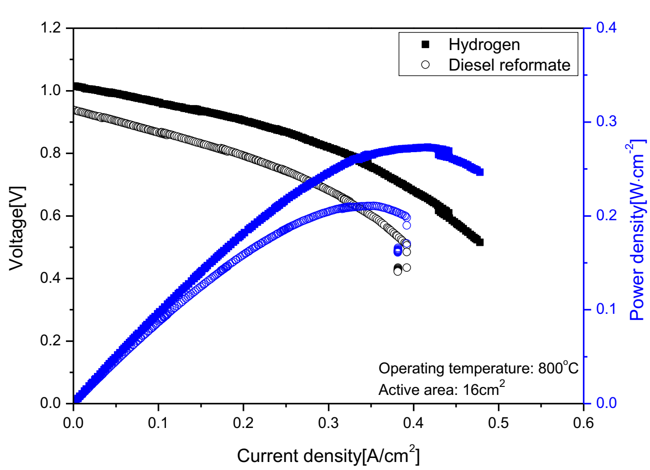

Fig. 2. *I*–*V*–*P* curves of a single stack with area of 50-mm × 50-mm using hydrogen and diesel reformate as fuel.

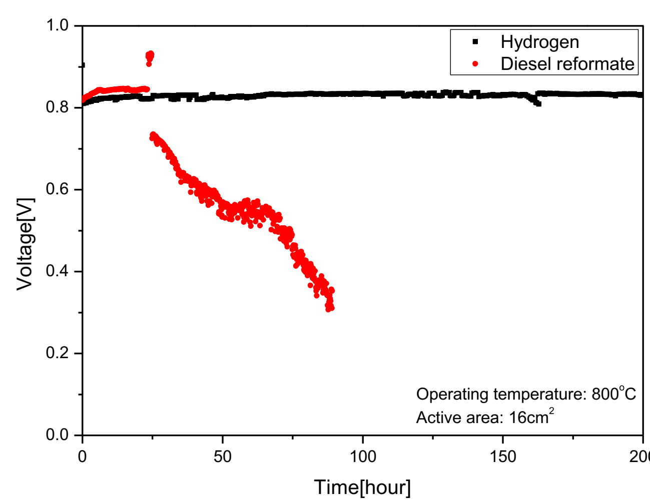

Fig. 3. Long-term operation of a single stack with area of 50-mm × 50-mm using hydrogen and diesel reformate as fuel.

Table 2
Thermodynamic calculation of oxygen partial pressure for metal oxidation.

$2Fe(s) + O_2(g) = 2FeO(s)$					$2Ni(s) + O_2(g) = 2NiO(s)$				
T (°C)	ΔH (kJ)	ΔS (J K^{-1})	ΔG (kJ)	log(P_{O_2}) (atm)	T (°C)	ΔH (kJ)	ΔS (J K^{-1})	ΔG (kJ)	log(P_{O_2}) (atm)
600	−527.434	−129.180	−414.641	−24.8	600	−472.498	−175.459	−319.296	−19.1
650	−527.808	−129.595	−408.173	−23.1	650	−471.950	−174.848	−310.539	−17.6
700	−528.535	−130.360	−401.675	−21.6	700	−471.413	−174.282	−301.811	−16.2
750	−529.755	−131.580	−395.129	−20.2	750	−470.889	−173.757	−293.110	−15.0
800	−530.871	−132.651	−388.517	−18.9	800	−470.378	−173.269	−284.434	−13.8
850	−531.181	−132.934	−381.876	−17.8	850	−469.880	−172.815	−275.782	−12.8
900	−531.219	−132.968	−375.228	−16.7	900	−469.394	−172.392	−267.152	−11.9

and the diesel reformate is obtained directly from the integrated fuel processor. The open circuit voltage (OCV) under the hydrogen operating condition is measured as 1.015 V, and that under the diesel reformate operating condition is measured as 0.938 V, as shown in Fig. 2. The gap between OCV values illustrated the different oxygen partial pressures in each fuel condition. The maximum power densities are identified as 0.27 W cm^{-1} and 0.21 W cm^{-1}, respectively, which indicates that the characteristics of diesel reformate operation have an influence on the SOFC's performance. To investigate the operating stability under each condition, the single stack is operated in a galvanostatic mode, in which a current density of 0.1 A cm^{-1} is applied to a single stack. Fig. 3 shows that the operation using diesel reformate shows serious performance degradation at a rate of 0.616%/hour, although the operation using hydrogen operated stably for 200 h.

The results indicates major differences in operating stability between the operations using hydrogen and the reformate, the key factor in which is the different fuels supplied to the anode. The critical characteristics of the diesel reformate operation are (1) low fuel concentration and (2) large amounts of steam. These characteristics are related to the reforming conditions required for stable operation, OCR = 0.8 and SCR = 3. Relatively high oxygen to carbon and steam to carbon ratios are needed for the diesel reformer's stable operation; thus, the reformate has high percentages of nitrogen and steam, which leads to high oxygen partial pressure. This high pressure may have induced the oxidation of the metallic component on the anode side. In particular, the design of the single cell is a metal-based SOFC, and the metal oxidation could have been more critical than in other types of SOFC. The possibility of metal oxidation due to high oxygen partial pressure in the fuel can also occur in other SOFC types because they have metallic components in their cells. The main components of a single cell that could be affected by the metal oxidation are iron and nickel in the anode. Thermodynamically, the oxygen partial pressures are calculated for each condition in terms of operating temperature and steam quantity when nickel and iron had oxidized, as shown in Table 2, and the results are calculated using Equations (1)–(3). The calculated results are plotted in the ternary diagram shown in Fig. 4. The logarithm of the oxygen partial pressure of the diesel reformate at 800 °C is between −17 and −19. As a result, the iron could be oxidized under the diesel reformate condition even when the steam is completely removed, although the nickel is safe enough for oxidation with any amount of steam. The operating temperature should be lower, and the calculation result at 750 °C illustrates the possibility of finding a reformate condition for stable operation. The logarithm of the oxygen partial pressure of iron oxidation at 750 °C is −20, and that of the diesel reformate is between −19 and −21, which means that the iron oxidation could be prevented if the steam quantity is controlled below 10%. A steam quantity of 5% is selected for the modified reformate condition, and this modified condition is validated using temperature-programmed reduction (TPR) analysis from a metal oxidation viewpoint. It is clear that the oxidation of powders such as AISI 410 and Ni is much more active than that of bulk metal, which means that the functional interface layer in the cell is more critical to oxidation than the metal support or interconnect. To check for oxidation tendency, the functional interface layer is charged in the reactor and exposed to the hydrogen (reformate) conditions. The synthetic gas replaces the diesel reformate with water supplied using a pump and vaporizer. The experiments are conducted under three conditions: (1) the bubbled hydrogen, (2) the synthetic gas containing 32% steam (identical to the diesel reformate), and (3) the synthetic gas containing 5% steam (modified conditions to prevent metal oxidation). The samples are exposed to each condition at 750 °C for 100 h and then analyzed using the TPR technique, the notable results of which are shown in Fig. 5. The large reduction peak occurs under the diesel reformate condition, although there is no significant reduction peak under the hydrogen or modified (synthetic gas with 5% steam) conditions. The modified condition does prevent metal oxidation, especially iron. Finally, the results of the thermodynamic calculation are validated by TPR analysis, and the

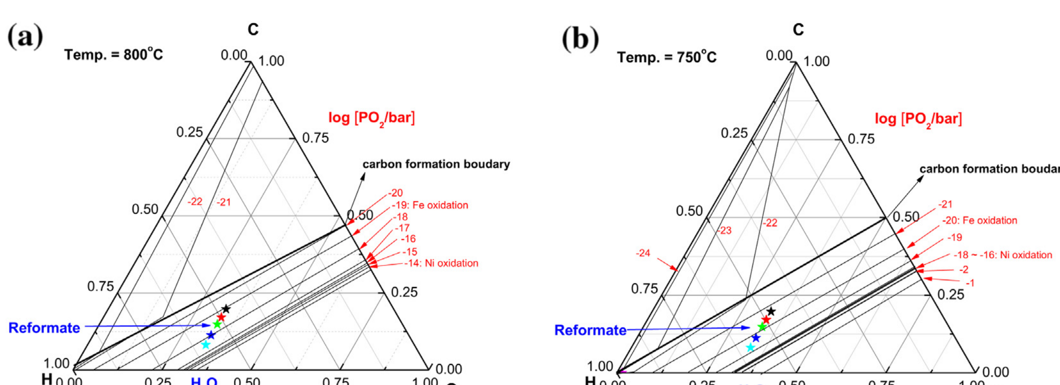

Fig. 4. Ternary diagram of oxygen partial pressure at (a) 800 °C and (b) 750 °C; the amount of steam in the fuel is cyan: 0%, blue: 5%, green: 10%, red: 20%, and black: 32%. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

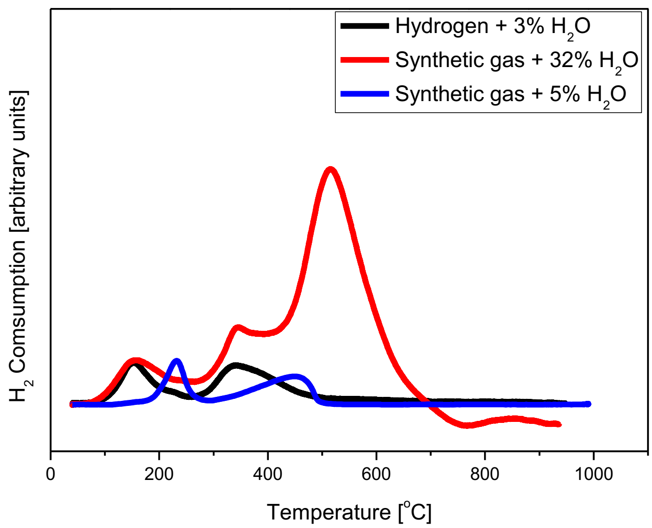

Fig. 5. TPR results for the functional interface layer exposed to the operating conditions.

cell operation is necessary to verify whether the stable operation is possible under the modified condition.

$$2Fe(s) + O_2(g) = 2FeO(s) \tag{1}$$

$$2Ni(s) + O_2(g) = 2NiO(s) \tag{2}$$

$$P_{O_2} = \frac{1}{a_{Fe}^2}\exp\left(\frac{\Delta G}{RT}\right) \tag{3}$$

$$OCV = \frac{RT}{4F}\ln\left(\frac{(P_{O_2})_{cathode}}{(P_{O_2})_{anode}}\right) \tag{4}$$

The button cell is fabricated with an active area of 0.785 cm^2 and subjected to both the hydrogen and the synthetic gas with varying amounts of steam. Fig. 6 shows the OCVs and *I–V–P* curves observes as the amount of steam was controlled to measure 0, 5, 10, 20, and 32%. The OCVs under the different conditions are calculated using Equation (4), based on Nernst equation, and the calculated and measured values are compared in Table 3. The OCVs are measured differently under each condition due to the different oxygen partial pressures, and the maximum power densities at each condition are observed differently. The maximum power

Table 3
Open circuit voltages in terms of gas and amount of steam.

750 °C	H_2	Synthetic gas				
	3% H_2O	0% H_2O	5% H_2O	10% H_2O	20% H_2O	32% H_2O
Measured	1.087	0.965	0.936	0.931	0.920	0.896
Calculated	1.111	0.998	0.983	0.970	0.949	0.928
Error	−2.17%	−3.28%	−4.77%	−3.98%	−3.09%	−3.46%

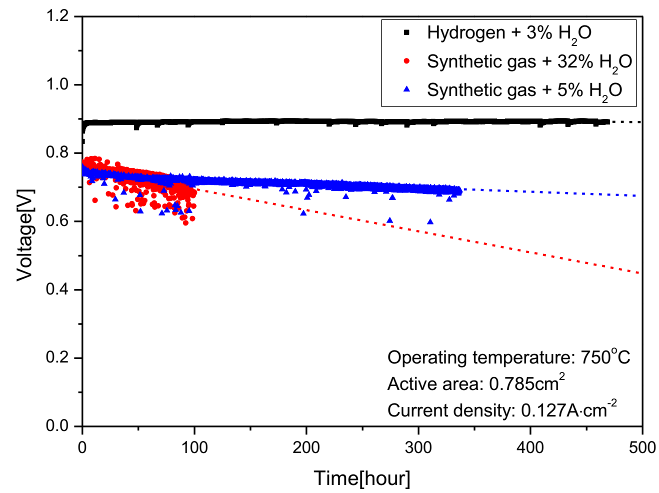

Fig. 7. Long-term operation of a button cell at 750 °C using hydrogen and synthetic gas with different steam quantities.

density decreases when the amount of steam increases, which illustrates how amount of steam affects the SOFC's electrochemical performance. These results are also evidence that the synthetic gas is successfully simulated by the diesel reformate.

Fig. 7 shows the long-term operation of the button cells under different conditions. The operation using hydrogen is stable for 500 h without any degradation, although there is a severe performance decline when the synthetic gas containing 32% steam is used as fuel. The operation using the modified condition, with synthetic gas containing 5% steam, is significantly improved. This result means that the fuel's oxygen partial pressure, which depends on the steam quantity, affects the operating stability. It is concluded that the fuel's oxygen partial pressure is one of the most important factors affecting the stable operation of a fuel cell system, especially a metal-based SOFC. A single stack operation is required to verify this result, so a SOFC single stack with an area of 50-mm × 50-mm is operated under the modified condition. This single stack is successfully operated for over 1000 h with a degradation rate of

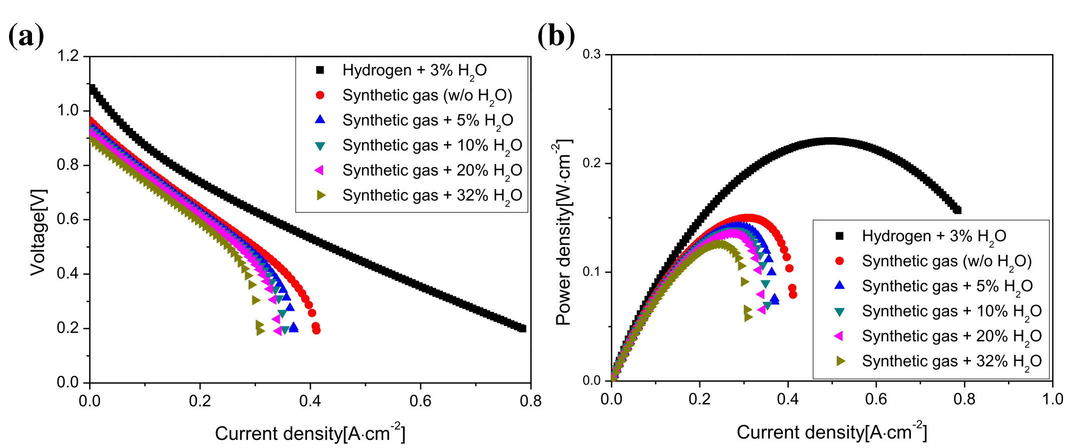

Fig. 6. Electrochemical performance of button cells at 750 °C in terms of gas and amount of steam: (a) *I–V* curves and (b) *I–P* curves.

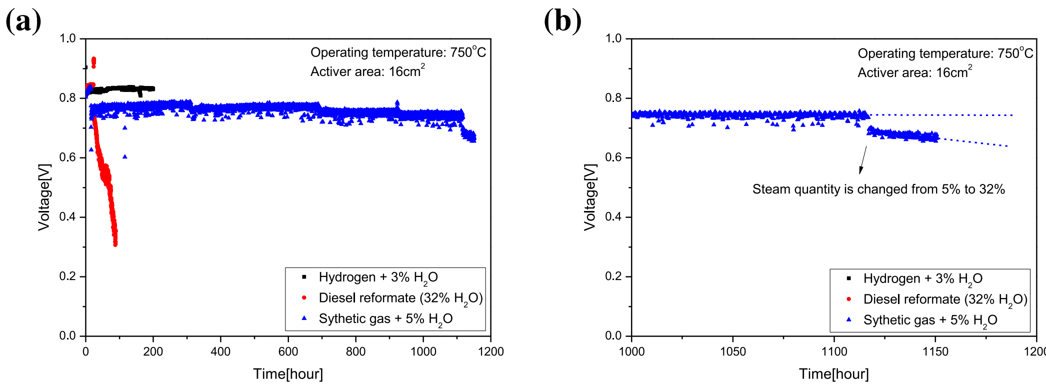

Fig. 8. (a) Long-term operation of a single stack with area of 50-mm × 50-mm under the modified conditions for 1000 h and (b) operation under the changed steam quantity after 1000 h of stable operation.

approximately 4%, as shown in Fig. 8(a). The graph shows the long-term operation under the three different fuels and indicates a significant improvement of stability when the modified conditions are used. Before the end of the test, the steam quantity is changed from 5% to 32% to verify its effect. It becomes clear that the degradation commenced with the feeding of 32% steam, as shown in Fig. 8(b).

4. Discussion

In this paper, the issues are claimed when metal-based SOFC is operated with diesel reformer. Diesel is basically difficult to convert the usable fuel for SOFC, and ATR is the most promising technology. Diesel reformate has high amount of steam and nitrogen, and the diesel reformate finally has relatively low fuel concentration and high oxygen partial pressure. This characteristic can severely affect the performance of metal-based SOFC. Low fuel concentration may give lower power performance of metal-based SOFC and high oxygen partial pressure can cause metal oxidation at SOFC components. These phenomena are observed at co-operation of metal-based SOFC and diesel reformate in this paper and proved by thermodynamics, TPR analysis, and measurement electrochemical performance. We suggested the modified operating condition based on these analyses, and the modified condition is reducing the operating temperature as 750 °C and the amount of steam as 5%.

The suggested operating temperature of 750 °C is an average value and there must be temperature distribution [31]. It is suggested that this temperature is a limitation for stable operation under the cell condition in this paper, and it is possible to be decrease the operating temperature but the maximum power drop is unavoidable. To solve this problem, the material for better performance at lower operating temperature is required to develop.

In the view of the steam amount, the present technology of ATR cannot give the solution unless the reforming method is changed to POx. Reducing the amount of steam in the reformate can achieve using a heat exchanger in the system. However, adding a heat exchanger cause loss of heat balance and can affect negatively total system efficiency. It is required to study carefully the method of reducing amount of steam with minimizing loss of heat balance from the efficient system point of view.

5. Conclusion

This study discussed the system operating conditions for metal-based solid oxide fuel cells using diesel reformate in terms of operating temperature and amount of steam in the fuel. Diesel reformate operation has specific characteristics, such as low fuel concentration and high steam quantity, due to its fuel processing conditions. These characteristics led to high oxygen partial pressure at the anode side and the oxidation of metallic components in the cell. Various approaches, such as thermodynamic calculation, TPR analysis, and the electrochemical performance of a single cell, were used to prove the oxidation phenomenon. Therefore, it was concluded that the operating condition of a diesel-driven metal-based SOFC should be modified such that the operating temperature and steam quantity in the fuel would be decreased. Using these results, a metal-based SOFC single stack with an area of 50-mm × 50-mm laser-welded to an interconnect to increase sealing efficiency was successfully operated for 1000 h with a degradation rate of 4%/1000 h under the modified condition. This operating condition should be considered for the stable long-term operation of a diesel-driven SOFC based on its fuel composition, oxygen partial pressure, and temperature. These results suggested practical and critical issues and solutions that could be considered for a highly durable and stable solid oxide fuel cell.

Acknowledgments

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